

REMARKS

This application has been carefully studied and amended in view of the Office Action dated March 25, 2009. The following comments incorporate matters discussed by the undersigned attorney with Examiners Katakam and O'Sullivan during the course of a personal interview on June 24, 2009. Examiners Katakam and O'Sullivan are respectfully thanked for the courtesies and suggestions made during the interview.

It is respectfully submitted that Claims 1-3, 5-8, 10-17, 19-39 and 41-44 are patentable over the prior art and in particular over Hara, et al. in view of Kaibel. Of these pending claims there are two independent claims, namely, Claim 1 and Claim 31.

The present invention relates to distillatively separating mixtures, specifically ethylenamine mixtures obtained by reacting monoethanolamine with ammonia wherein ammonia and water are removed leaving an ethylenamine mixture of EDA, PIP and other components, such as DETA, AEEA and/or MEOA. To this point the process is similar to Hara, et al. As recognized by Examiner Katakam the invention departs from Hara, et al. in using dividing wall columns (Claim 1) or thermal installations (Claim 31) for removing the ethylenamine components.

As regards the use of dividing wall columns (DWC), Claim 1 as now amended, defines high purity, high color quality EDA to be removed as a top product from the DWC and high purity, high color quality PIP to be removed as a side draw product. The operating pressure in the DWC is 0.1-5 bar. Significantly, the EDA and PIP are simultaneously removed from the same DWC.

As recognized in the Office Action and noted above, Hara, et al. do not disclose using dividing wall columns for the distillation to separate the ethylenamines. Accordingly, there is reliance on Kaibel for that feature. Kaibel relates to distillation columns with vertical partitions and discloses the separation of a feed mixture into three or four or more pure fractions. Kaibel does not, however, disclose the utilization of DWCs for separating ethylenamine mixtures and

particularly where there is a simultaneous separation of the components EDA and PIP from the same DWC. To obtain components in high purity from a single column is a challenging task as based on PIP's high melting point. There is a significant risk of blockage of the column internals by solidification of PIP. In the conventional process where EDA and PIP are produced from two distinct columns, this problem of blockage scarcely occurs. This is due to the fact that in the conventional process operating pressures and temperatures can be independently chosen in separate columns, whereas in a DWC a compromise has to be found.

As pointed out in the Specification, at page 10, lines 17-20, it is preferable in the purifying distillation of EDA and PIP to operate "at pressures slightly above atmospheric pressure so that the temperature in all regions of the column is slightly above the melting temperature of PIP..." Since Kaibel does not relate to separating ethylenamine mixtures, there likewise is no disclosure in Kaibel of using an operating pressure of 0.1 to 5 bar for the separation, as defined in Claim 1. Thus, Kaibel is devoid of any teaching that would assist one of ordinary skill in the art as to what techniques and operating conditions should be used so that it would be feasible, and therefore obvious, to simultaneously separate EDA and PIP from the same DWC.

The fact that DWCs are suitable for the separation of multi-component mixtures does not mean that they would be applicable to any and all mixtures irrespective of the substances involved and their respective physico-chemical properties. Furthermore, despite the known advantages, the use of DWCS restricts the flexibility for operating a distillation and introduces higher complexity to the system. Thus, for this reason alone a person of ordinary skill in the art would not automatically resort to the DWC technology even with knowledge of Kaibel in order to separate multi-component mixtures.

The prior art approach to separating EDA and PIP and obtaining those products in high purity and high color quality form is to use separate and distinct columns for each of the EDA and PIP. The advantages of DWC's were known. Kaibel refers to some advantages. Kaibel was known years before Hara, et al., yet Hara, et al. make no disclosure of using DWC's or thermal

installations for simultaneously removing both EDA and PIP from the same column or installation. The present specification points out that the same high quality EDA and PIP are obtained with the invention as with the prior art but the use of the invention “reduces the total number of distillation columns required in the separation of multisubstance mixtures” (page 1, lines 40-41) and “Dividing wall columns and thermally coupled distillation columns offer advantages compared to the arrangement of conventional distillation columns both with regard to the energy demands and the capital costs, and are therefore being used to an increasing extent in industry” (page 2, lines 8-11). Yet, it was not until the present invention that DWC’s (Claim 1) or thermal installations (Claim 31) could be used for ethylenamines where EDA and PIP are simultaneously removed from the same DWC or thermal installation.

While Kaibel recognized advantages of DWC in a generic sense, Kaibel does not teach or suggest its use for ethylenamines as with the claimed invention. Because of the particular characteristics of ethylenamines, its use in DWC’s and thermal installations, as defined in Claims 1 and 31, would not be obvious.

The use of DWCS is not what one of ordinary skill in the art would readily think as being obvious to resort to. In that regard, mixtures comprising amines are known to pose problems where separation is sought. See, for example, Riechers U.S. 6,627,756 which relates to the preparation of pure triethylenediamine (TEDA) by vaporizing TEDA and introducing the gaseous TEDA into a liquid solvent. As stated in Riechers “Owing to its properties...TEDA can be handled only with difficulty and with an appropriate engineering outlay if deterioration in the quality of the TEDA...is to be avoided”. (Col. 1, line 65 to Col. 2, line 9) This statement is also true for the mixture in question in Claims 1 and 31. In particular, a solution for the problem of separating the mixture into components, each of which should be of high purity and high quality was sought. At least for the components EDA, PIP, DETA and AEEA “high purity” means a respective value of more than 99.0% by weight. (See pages 3-5 of the present Specification) Specifically, the problem of color improvements was to be solved as pointed out on page 2, lines 24-28 of the present Specification. Neither Hara, et. al, nor Kaibel, nor any other prior art relating to the general state of the art of distillation, teaches the separation of the mixture into the

components according to Claims 1 and 31 to produce various products with high purity and high color quality.

Attached is a copy of a publication by Becker et al (Chemical Engineering, January 2001, pages 68-74) which focuses on some challenges when designing a DWC. Page 70 includes the discussion under the heading "Not a panacea". Note is also made of attached publication by Schultz et al. (Chemical Engineering Progress, May 2002, pages 64-71) which presents a general background of DWC technology and at page 66 specifically raises cautions in its discussion of "The case against DWCS". Thus, these publications raise caution to the conclusion that by simply having knowledge of Hara et al and Kaibel it would have been obvious to provide a method such as defined in Claim 1, wherein EDA and PIP are simultaneously removed from the same DWC.

In view of the above, Claim 1 and its dependent claims should be allowed.

Claim 31 is similar to Claim 1 except that instead of using DWCS for the removal of EDA and PIP, a thermal installation in the form of a connection of two distillation columns is used wherein EDA is obtained as a top product and PIP as a side draw stream. Both the EDA and PIP are simultaneously removed from the same connection of the two thermally coupled installation columns. As recognized in the Office Action Hara, et al "is silent on thermally coupled distillation columns". Although Kaibel shows in Figure 2 thermally coupled distillation columns, there is no suggestion in Kaibel of utilizing thermally coupled distillation columns for separating ethylenamine mixtures and in particular that EDA and PIP can be simultaneously removed from the same connection of the columns as defined in Claim 31. Accordingly, Claim 31 and its dependent claims should be allowed.

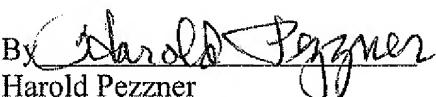
In summary, the prior art does not disclose or teach the simultaneous separation of pure EDA and pure PIP from the same DWC. Rather, because of the awareness that, for example, DWC's were not a panacea and the belief that sometimes DWC's were not even feasible for some mixtures, the prior art considered the conventional distillation sequence as the better (or

only) alternative. The unobviousness of the present invention is clear from the question: Where is there any prior art teaching the simultaneous separation of pure EDA and pure PIP from the same DWC or the same thermal installation? The fact that there is no such prior art establishes the patentability of the claims.

In view of the above remarks and amendments this application should be passed to issue.

Dated: June 25, 2009

Respectfully submitted,

By 
Harold Pezzner

Registration No.: 22,112
CONNOLLY BOVE LODGE & HUTZ LLP
1007 North Orange Street
P.O. Box 2207
Wilmington, Delaware 19899
(302) 658-9141
Attorney for Applicant

Partitioned Distillation Columns – Why, When & How

Hans Becker, Sven Godorr
and Helmut Kreis
Linde AG.

James Vaughan,
Sasol Technology

Because of the significant advantages that they offer in certain applications, partitioned columns allow a topping and tailing step to be accomplished in a single distillation column

Engineers in the chemical process industries (CPI) are giving more and more attention to a type of distillation column, called a partitioned column, that contains a vertical baffle in its middle section. From a single such column, three essentially pure product streams can be withdrawn. In other words, a topping step (recovery of a desired low-boiling component) and a tailing step (recovery of a desired high-boiling component) can be accomplished at the same time in the same tower, while furthermore removing the intermediate-boiling product as a sidestream.

In such a column, also referred to as a divided-wall or Pettyuk column, the vertical baffle is placed so as to separate the feed location from that at which the intermediate-boiling product is withdrawn. In this way the amount of heavy (high-boiling) or light (low-boiling) components reaching the side draw may be controlled to any specification.

Partitioned columns have been studied for several years (box, p. 74), as have thermally coupled columns,* which approximate the operation of a partitioned column. However, both have only recently enjoyed increasing attention, as engineers become more confident about being able to design and operate them properly.

Being able to carry out topping and tailing steps within a single column obviously offers capital-cost savings.

*Thermally coupled columns consist of two (or sometimes more) columns that are linked together by their overhead and bottoms streams without intervening reboilers or condensers.

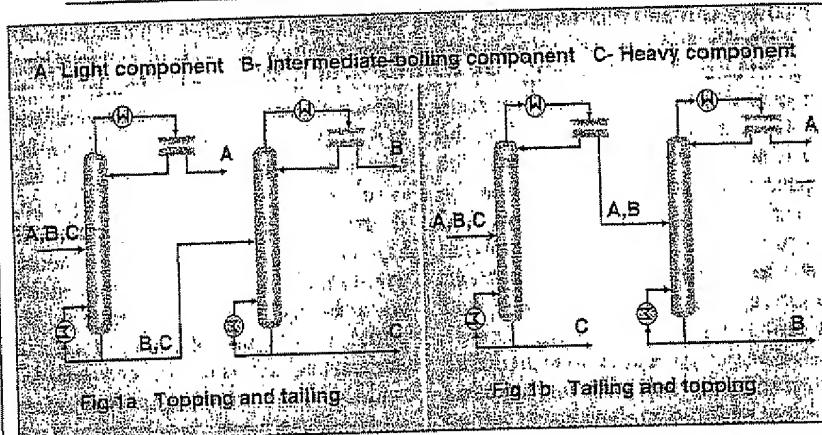


FIGURE 1. For separating three components, conventional fractionation requires at least two columns. Schemes that are more energy-efficient appear in Figure 2.

Further, the thermal coupling of the fractionation steps provides thermodynamic benefits that significantly reduce the heat duty required for a given separation. Thus, the use of partitioned columns can lower both the fixed and the operating costs for achieving a given separation.

This article offers guidelines for assessing whether a partitioned column is suitable for a particular application. It also provides the basis for developing a preliminary design for such a column. Furthermore, it highlights critical issues and pitfalls that should be kept in mind with respect to the optimization and control of such systems.

Setting the stage

To lay the groundwork, we first look at conventional distillation technology. Normally, the separation of a process stream into low- (A), inter-

mediate- (B), and high-boiling (C) fractions is accomplished in successive topping and tailing steps (Figure 1a): The light component (A) comes off the first distillation column, and the intermediate and high-boiling components (B and C) are then separated in a second column.

This sequence is generally considered to be less energy-intensive than conducting the tailing step first (Figure 1b), because in Figure 1a, the light component is only boiled off once. However, it is preferable to conduct the tailing step first under certain circumstances; for example, if the amount of high-boiling material to be removed is relatively small.

From the standpoint of thermodynamics (and, accordingly, energy efficiency), the optimal conventional distillation arrangement for this separation requires not two but three columns. The light and heavy

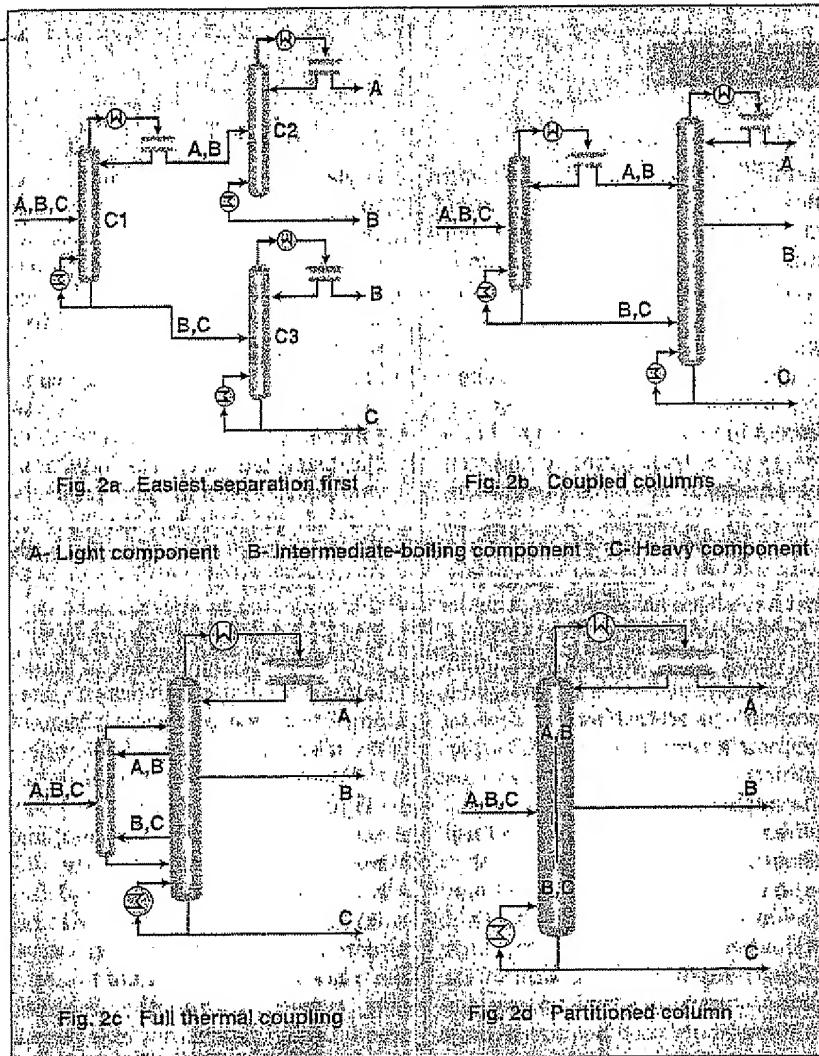


FIGURE 2. For the energy-efficient separation of three components, this sequence of four options leads logically to partitioned columns (2d) as being the simplest of them

components should be separated in an initial step, as shown in Figure 2a. Here, the intermediate-boiling component appears in both the overhead and bottoms streams of Column C1, before it is separated from the light and heavy components in two further columns, C2 and C3.

Although it is thermodynamically more attractive than the arrangements in Figure 1, the setup in Figure 2a does require an additional column. By combining C2 and C3 into a single column, as shown in Figure 2b, the engineer can achieve the identical separation in two linked distillation columns. This reduces the number of required equipment items. Even though each column has its own reboiler and condenser, the system has a better thermal efficiency than the arrangement given in Figure 2a, thanks to the reuse of the vapor generated in the reboiler of

what was previously Column C3, in separation stages that were previously contained in C2.

A further integration step is shown in the simplified arrangement given in Figure 2c. Here all the required stripping vapor and reflux are generated in a single reboiler and condenser. Meanwhile, a portion of the flow is transferred to and from the column into which the feed is introduced. This is a full thermal coupling of the separation processes in C1, C2 and C3.

In the final simplification, these process steps can instead be accomplished within a single distillation column by using a partition, as shown in Figure 2d. On the feed side of the partition, a separation is achieved between the light and heavy fractions, while the intermediate-boiling fraction is allowed to migrate to both the top and bottom of the partition.

On the product side of the partition (the side not facing the feed), the light components are stripped overhead and the heavy components are washed down. This allows the desired purity of the intermediate-boiling fraction to be achieved.

In light of the progressive integration and simplification shown in Figure 2, the partitioned column is the ordinarily optimal design. Nevertheless, in certain revamp situations, the coupling of columns as shown in Figure 2b, with a suitable choice of internals, can lead to substantial increases in separation capacity. In some other cases, where existing equipment is to be reused, it may also be sensible to consider an arrangement such as that given in Figure 2c.

While the advantages of a partitioned column may be most easily illustrated by means of such a conventional-fractionation example, this technology may be equally well applied to extractive or azeotropic distillation systems.

Fully coupled columns (Figure 2c) and partitioned columns (Figure 2d) are thermodynamically equivalent. Accordingly, most of the concepts and results presented in this paper are directly applicable to both.

Partitioned columns' pluses

As already pointed out, combining topping and tailing steps into a single vessel allows the engineer to avoid the need for an entire distillation column, as well as its associated equipment (reboiler, condenser, reflux drum, reflux and bottoms pumps) and instrumentation. Admittedly, the partitioned column and its equipment are larger than either the otherwise-required topping or tailing column, but the overall capital investment is usually significantly lower than for a two-column system. For new-plant projects, capital-cost savings of around 30% are not unusual.

As for partitioned columns' thermal benefits, the separation uses the same total number of theoretical stages but involves significantly smaller reboiler and condenser duties than two-column arrangements. Reductions of about 30% in total reboiler duty are typical, but in some cases the

overall lowering of condenser and reboiler load can reach 50%.

In partitioned columns, the process stream is only reboiled once, and its total residence time in high-temperature zones is minimized. This aspect is especially valuable if heat-sensitive components are involved — there are many situations in which compounds formed by thermally induced degradation can adversely affect the quality of the product streams. For instance, the unintended formation of heavy polymers can harm the bottoms product, while also leading to excessive reboiler fouling.

Not a panacea

Partitioned columns are not the optimal solution for every fractionation task that involves a topping and tailing step [1, 2]. To understand this limitation, it must be realized that the thermodynamic benefits of a partitioned column are partly due to the "reuse" of the heat input throughout the column.

Specifically, the vapor generated by the reboiler serves first to strip the intermediate-boiling components from the heavy fraction, and then to lift the low-boiling components overhead. Ideally, the internal flow must be present along the full length of the column. This requires all the reboil to occur in the column sump, at the highest temperatures in the column, while the reflux must be generated at the very top of the column, where the temperatures are the lowest. Thus while the amount of energy that is required is minimized in terms of the first law of thermodynamics, the energy must nevertheless be provided at the highest temperature level and rejected again at the lowest temperature level.

Either or both of those requirements can become problematic if a relatively expensive heat or cooling source must be used in the reboiler or the condenser, or if the recovery of useful heat from the condenser is no longer possible.

Particularly in cryogenic applications, or others where refrigeration is required for the condensation, the cost of energy rejection becomes sub-

stantial. In these cases, it becomes important to minimize the amount of energy to be removed at low temperatures. Similar considerations apply to the reboiler if higher-pressure steam or a fired heater is required to achieve the reboil.

Admittedly, the use of side reboilers, intermediate condensers or similar equipment may allow part of the energy to be transferred to and from the process at more-reasonable temperatures. However, the need for this additional equipment offsets some of the capital-cost advantages that have been achieved in opting for a partitioned column. Furthermore, the contribution of such equipment to the internal flow is not available on all the theoretical stages.

Other factors that may make partitioned columns unsuitable for certain applications are:

- **Size:** Every partitioned column has more theoretical stages than the either of the two conventional columns that it has superseded. (However, it will always have fewer stages than the sum of the two columns.) If the topping and tailing steps each require a large number of stages, the result may be a very tall partitioned column, which may approach mechanical limitations as regards manufacture and erection.
- **Metallurgy:** If either the superseded topping or tailing column would require a relatively expensive material of construction (for instance, to assure product purity), this material will have to be specified for the corresponding sections of the partitioned column. This requirement may make the partitioned column so expensive that it is cheaper to build two smaller columns, where only one makes use of the more-noble metallurgy.
- **Operating pressure:** In a partitioned column, both the topping and tailing separations take place at about the same pressure (neglecting the pressure drop within the column). If one of those separations requires vacuum conditions to enhance the relative volatilities of certain components whereas the other separation can be easily

achieved at a higher pressure, it may be more cost-effective to use two separate columns operating at different pressures. (Limited opportunities exist for thermally coupling columns operating at different pressures.)

- **Hydraulic imbalance:** The composition and properties of the process streams on the two sides of the partition are, by definition, somewhat different. If a property such as the foaming tendency of the mixture varies strongly with the composition of the streams, this may lead to unpredictable hydraulic behavior on one side of the partition. The importance of maintaining a balanced hydraulic load on either side of the partition is discussed in more detail below.

Design and simulation

As with all distillation design and simulation problems, accurate description of the vapor-liquid equilibrium is essential. Optimal design also requires a good description of the concentration profiles of the various species within the column, since these determine how these species move around the partition from its feed side.

Before simulating a partitioned column, the engineer will find it useful to develop a preliminary design for the conventional two-column system that would be required to achieve the same separations. This activity provides a base case against which the partitioned column can ultimately be compared, and helps the engineer develop an understanding of the required separations.

As a first estimate for the number of theoretical stages needed in the equivalent partitioned column, try 80% of the sum of the number in the two columns. For instance, if 20 theoretical stages are required in the one conventional column and 30 in the other, we recommend that the initial simulations of the partitioned column use about 40 stages. Initially, the partition should be positioned in such a way that it is opposite the middle third of the equilibrium stages.

For the initial simulations, we rec-

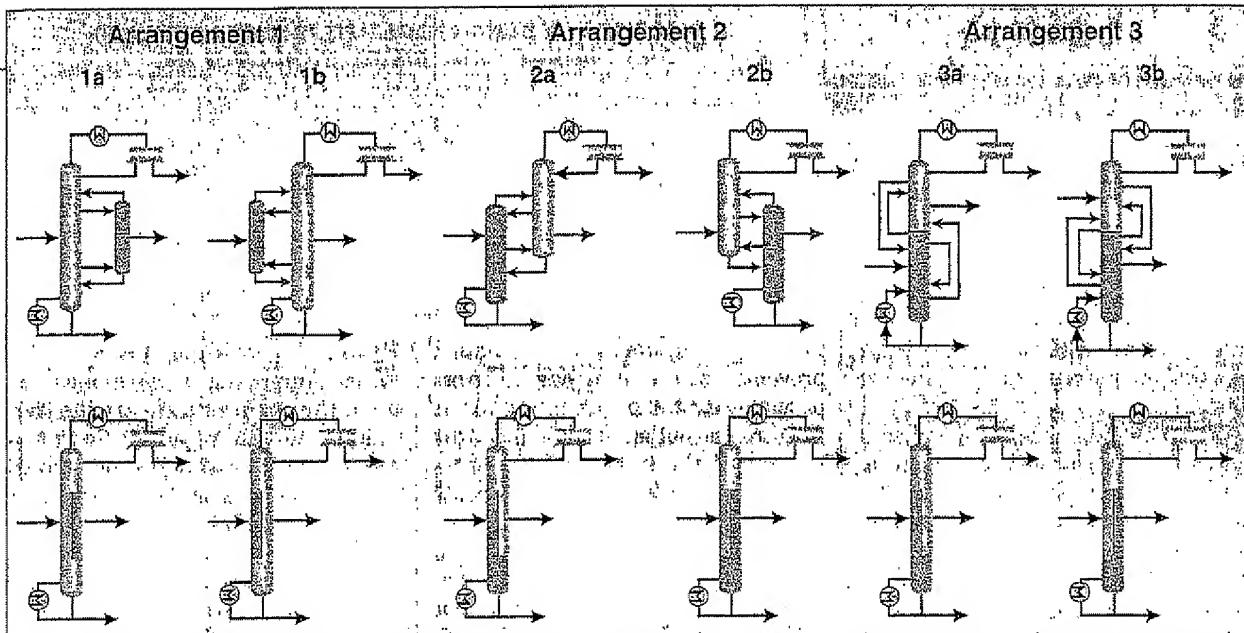


FIGURE 3. Most simulation packages available today do not routinely accommodate partitioned columns; instead, the engineer must turn to sequences of conventional columns. In each of the six vertical panels of this figure, the shaded section in the upper, conventional column sequence corresponds to the shaded portion of the partitioned column that appears immediately below.

ommend that the internal flow in the column (as denoted by the reboiler or condenser duty) be set to about 70% of the total duty for the two individual columns. Thus if the reboiler duties of the two conventional columns were 3 MW and 2 MW, the partitioned column should initially be given a reboiler duty of about 3.5 MW. For a first-round simulation, the vapor and liquid flow should be split equally between the two sides of the partition.

In most simulation packages, partitioned columns are currently not available as standard unit operations. To simulate a partitioned column, it is usually necessary to couple two conventional distillation units thermally, so as to define the equivalent separation problem. In Figure 3, various possible arrangements of coupled conventional columns are shown in the upper row, each above the equivalent partitioned column. In each pair, the shaded section in the simulated flowsheet corresponds to the shaded portion of the partitioned column shown immediately below.

The simulation represented by Arrangement 2 is particularly interesting, because it lets two columns be fully coupled, thermally, in a way that the lower section can be operated at a somewhat higher pressure than the upper part of the column.

Depending on the particular separation task, column-simulation algorithm and recycle-convergence method, the various flowsheet alternatives can exhibit quite varied be-

havior as regards overall convergence.

In Figure 3, Arrangements 1 and 2 each make use of two distillation columns, so conventional, sequential modular simulators can solve these systems, iteratively. Be aware, however, that these simulations may exhibit poor convergence because of the strong interaction between the units; and in large columns containing complex mixtures, the computational costs can become excessive.

Despite those two cautions, Alternative 1b often proves to be a convenient arrangement for sequential modeling, because it allows specifications to be set for all three products, while the free (independent) parameters in the same unit can also be varied.

Arrangement 3 in Figure 3 assumes the use of pumparounds and vapor bypasses, to simulate a partitioned column in a single "conventional" distillation unit. This arrangement, however, requires complete liquid and vapor withdrawal to be taken at two points in the column (that is, no liquid remains to descend to the tray below, and no vapor remains to arise to the tray above), which can lead to convergence problems. On the other hand, since this arrangement requires only the simulation of a single unit to be solved, it represents a comparatively efficient way to get a solution for a given system.

Equation-based simulators, which directly and simultaneously solve

the entire flowsheet, offer significant advantages over sequential modular simulators for dealing with partitioned columns, because the recycle loops associated with a two-column setup or with various controllers are eliminated. This greatly enhances the convergence stability and speed.

Optimizing the columns

Before discussing the optimization of a partitioned column, we review here the well-understood design and optimization of a conventional distillation column. For this discussion, a conventional column is one with a single feed, and with the products being withdrawn as overhead and bottoms streams. Numerous heuristic rules are available for such systems, and commercial simulators offer useful tools for performing many of the required optimizations and sensitivity analyses.

Conventional columns: Generally, the first step in designing such a column is to set its operating pressure. This task involves factors such as the flare-header back pressure, the availability of utilities for heating the reboiler and cooling the condenser, the thermal stability of the species and the effect of the pressure on the vapor-liquid equilibrium.

Once the pressure has been set, the design engineer chooses the optimal combination of internal flowrates and number of stages, to achieve the required separation in terms of product purity, recovery or

COMPARISON OF DESIGN PARAMETERS FOR PARTITIONED AND CONVENTIONAL DISTILLATION COLUMNS

	Conventional Column	Partitioned Column
Discrete (integer) parameters	Total number of stages Feedpoint location	Total number of stages Feedpoint location Side draw location Location of bottom of partition Location of top of partition
Continuous parameters	Total internal flow	Total internal flow Distribution of vapor Distribution of liquid

both. For a given separation task, various guidelines are available for finding the best point on the curve that relates the required internal flowrates (as denoted by reboiler duty, reflux rate or some other equivalent quantity) to the number of stages.

The only "sub-optimization" that needs to be performed for a conventional column is for choosing the optimal feedpoint location. This task is usually carried out simply by achieving the best match between the concentrations in the feed stream and the concentration profile in the column while taking the feed temperature into account. For multicomponent systems, the match is often conveniently made by varying the feedpoint to minimize the required internal flowrate for a given number of stages.

In such simple columns, only limited interactions occur between the parameters that are available to the design engineer. Therefore, it is often possible to accomplish an acceptable optimization by varying the parameters sequentially.

Partitioned columns: If a column is to be partitioned or coupled to another column, the number of design parameters and interactions between variables increases sharply. For instance, it is possible to vary both the length and the position of the partition, in addition to the location of the feed(s) and sidestream(s). Further the distribution of vapor and liquid on either side of the partition represent additional degrees of freedom.

As the partition is lengthened downward in the column, that device becomes more effective at preventing light species from slipping beneath and reaching the sidestream outlet directly. On the other hand, the intermediate-boiling fraction has more difficulty in reaching the sidestream outlet by passing below the partition. This hindrance either forces more of the intermediate component over the top of the partition, or raises the need for additional fractionation capacity below the partition to separate the intermediate- and high-boiling fractions.

Conversely, lengthening the parti-

tion too far upwards in the column prevents heavy components from passing over the partition top to the sidestream outlet, but makes the passage of the intermediate component to the sidestream via this route more difficult.

Other variables to be considered are the positioning of the feed stream and of the sidestream. These choices significantly influence the concentration profiles that are set up within the column.

The increased number of parameters, and the strong interaction between them, make it impossible to optimize such a system by varying one parameter at a time. It is instead necessary to consider all the parameters and their interdependence simultaneously, as well as the sensitivity of the objective function to any changes that are made.

In light of the various complications just discussed, it is not possible to optimize a partitioned column properly by relying just upon the experience, intuition and skill of the design engineer. Some work [3] has taken place on developing procedures for optimizing the design of partitioned columns. Results from the application of mixed-integer, non-linear programming algorithms to this problem have also been reported [4]. At any rate, carrying out high-quality optimizations of partitioned columns for typical process separations require suitable (equation-based) simulators that offer powerful optimization routines.

The parameters that are varied in an optimization are of two types. Some are integer values, such as feedpoints, which can only be varied in discrete steps. More prevalent are continuous variables, such as the reboiler duty (which is linked with the flowrates within the column). The table above compares the fundamental design variables, of both kinds, in a conventional distillation column design to those that must be considered in a partitioned column.*

Hydraulic considerations

When simulating a partitioned column, the engineer can vary the fraction of the total vapor traffic that is routed to either the feed or product side of the partition; and in principle, this variation may be included as part of the optimization exercise.

An asymmetrical distribution of the vapor traffic between the two sides of the partition may offer thermodynamic advantages in certain situations, depending on the conditions at which the feed is available or at which the product streams are required. Asymmetrical vapor distribution can, in principle, be achieved in two ways: The partition itself can be positioned off-center, or the internals can be designed so as to induce more pressure drop on one side than on the other.

Off-center positioning has been tried at a laboratory scale [5]. In most industrial units, however, the partition instead divides the column exactly in half, this choice being based on cost, mechanical stability, ease of fabrication, and good performance by the column internals. In these latter columns, the additional pressure drop on one side (so as to distribute the vapors asymmetrically) can be achieved either by means of incorporating a specifically designed restriction (whose dimensions might in some cases be controlled) or by designing the basic internals differently.

In either case, such an exercise requires very accurate pressure-drop correlations. Furthermore, it may bring about additional pressure drop through the overall column. Accordingly, the most-popular arrangement in practice is to have not only a centrally placed partition but also an even split of the vapor between the

*The employer of three of the authors (Linde AG) has developed an in-house equation-based simulation program, "Optim Design," in whose optimization routines it is possible to vary discrete and continuous variables simultaneously. The objective function for the optimization may be defined by the user to include a variety of factors, such as purity, energy consumption and capital cost, and the entire optimization may be subject to various constraints.

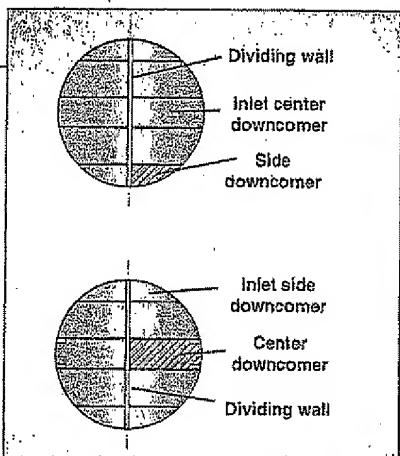
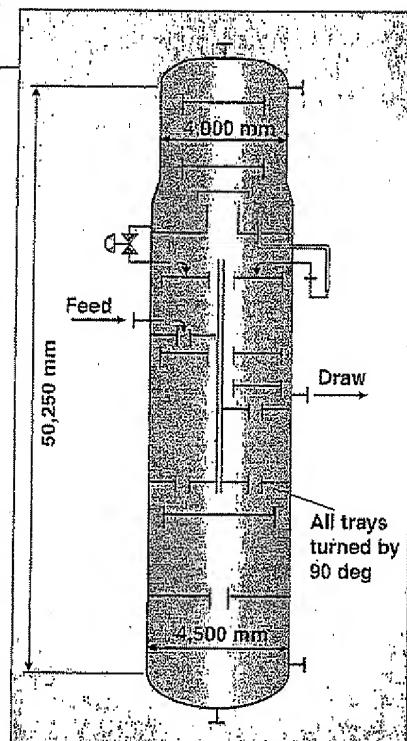
sol-
; is
uct
ole,
as

the
; of
dy-
ra-
at
ich
ed.
an,
ys:
ted
de-
re

en
In
he
nn
ng
ty,
br-
In
al
to
ri-
by
ly
n-
n-
n-

e-
op
ay
sp
d-
e-
a
in
ie

de
ed
se
is-
ly
sy
ic
nd
be



FIGURES 4 (left) AND 5. The internals of one existing partitioned column (see main text) appear on the left; shown above are details of its tray arrangement

two sides of the partition (and, thus, symmetric vapor flow).

As for the internal liquid reflux, its distribution between the feed and product sides of the column is a powerful design and control variable, which always has an optimal value for a given separation task. In practice, therefore, the vapor distribution is generally symmetric while the liquid distribution is asymmetric.

To bring about the desired column performance with the optimal flowrate of reflux on each side of the partition, it is essential that the vapor distribution occur as intended by the designer. This requires the internals to be specified, manufactured and installed in such a way that the pressure drop on either side of the partition can be equal for the desired vapor distribution.

Structured packing is generally used for partitioned columns. However, Linde has built three partitioned columns that are equipped with trays. In many applications, trays are preferred over packings for reasons of cost or hydraulics. For example, at high pressures (20 bar, say), the use of packings instead of trays can lead to poor vapor-liquid contact.

Two of these trayed partitioned columns are in operation at the Sasol fuels and petrochemicals complex in Secunda, South Africa. Currently the largest partitioned columns in the world, these are used to recover high-value petrochemicals from Fischer-Tropsch-based feedstocks. The third, much smaller, column forms part of a Rectisol wash that has been installed at a plant in the Far East.

Typifying large partitioned columns with trays, the first such column installed at Sasol has a total height of 64.5 m and a straight-side length of 50.25 m. It is equipped with 87 sieve trays. The vessel diameter is 4.5 m, which decreases to 4.0 m toward the top. Figure 4 is a schematic illustration, while Figure 5 provides details of the tray arrangement. The second, still larger, Sasol unit, having a diameter of 5.2 m and a tangent length of 99.3 m, has recently been brought onstream.

Both columns employ two-pass sieve trays. They were split in such a way that the partition is perpendicular (in a horizontal plane) to the downcomer walls. This orientation gives the best liquid flowpath on the trays, as well as additional mechanical stiffening of the partition by the tray rings.

The control challenge

As noted in the box (p.74), concerns about the control of partitioned columns [6, 7] probably contributed to the delay in the first commercial implementation of this technology. While the same fundamental principles apply for the control of partitioned columns as for conventional columns, certain characteristic differences must be considered because of the increased complexity.

Again, consider first the control of a conventional column. In such a column, the desired separation requires, in principle, that two independent control parameters be set. Key examples are the reboiler duty

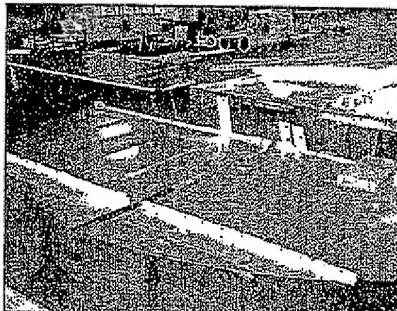


FIGURE 6. Here is a fabricator's view of a two-pass sieve tray that has been manufactured for a partitioned column

and the overhead product flowrate or the reflux ratio.

The measurable variables according to which these conventional-column parameters are adjusted can be, for example, the feed flowrate and a property analysis at either a suitable point in the column or in one of the product streams. In practice an actual chemical composition analysis is not usually used; it is more convenient to choose some other property uniquely related to the composition. In many cases, a temperature measurement is most suitable. In any case, even with the conventional columns, the control system should be adapted for each application.

Partitioned columns in effect have three more degrees of freedom than do conventional columns. These are the distribution of vapor flow and liquid reflux to either side of the partition, and the amount of material withdrawn as the intermediate product. Accordingly, the system is significantly more complex to control, and it is thus even less likely that a universally optimal control philosophy for partitioned columns could be defined. The control parameters and the associated measured variables must be chosen to suit the specific application.

Should theoretical considerations or analogies to other systems not provide a clear indication of how a column may be controlled best, a dynamic simulation will often provide useful assistance.

Final thoughts

Once a separation task has been identified as a candidate for partitioned-column technology, design should proceed through preliminary

Cover Story

simulations and sizing. Beyond that stage, there are several areas where input from contractors — or, if feasible, from operating companies that have experience with such columns — should be obtained:

- Optimizing the column design
- Specifying the internals, to ensure attention to issues such as heat transfer across the partition, maldistribution of vapor and liquid, and mass-transfer efficiencies in the partitioned section of the column
- Developing the correct control strategy, so that the column can be operated stably and will achieve all of the specifications

Finally, if a column is to be installed for the first time at a facility, it is important to ensure that the personnel who will operate it are involved throughout the development of the design, and that suitable training is provided. A proper understanding of a novel technology such as this is essential to develop the trust and skills required for its successful operation.

References

1. Agrawal, R., and Fidkowski, Z. T., Are Thermally Coupled Distillation Columns Always Thermodynamically More Efficient for Ternary Distillations?, *Ind. Eng. Chem. Res.*, 37, pp. 3444–3454, 1998.
2. Agrawal, R. and Fidkowski, Z. T., New Thermally Coupled Schemes for Ternary Distillation, *AICHE J.*, 45, No. 3, pp. 485–496, 1999.
3. Triantafyllou, C., and Smith, R., The Design and Optimisation of Fully Thermally Coupled Distillation Columns, *Trans. I. Chem. E.*, 70, Part A, pp. 118–132, 1992.
4. Bauer, M.H. and Stichlmair, J., Strukturansynthese und Optimierung nicht-idealster Rektifizierprozesse, *Chemie Ingenieur Technik*, 68, 8196, pp. 911–916, 1996.
5. Mutlib, M.I.A., others, Operation and Control of Dividing Wall Distillation Columns - Part 2 : Simulation and Pilot Plant Studies Using Temperature Control, *Trans. I. Chem. E.*, 76, Part A, pp. 319–334, 1998.
6. Wolff, E.A., others, Dynamics and Control of Integrated Three-Product (Petyluk) Distillation Columns, "I. Chem. E. Symposium Series No 133," pp. 111–118, 1996.
7. Halvorsen, I.J. and Skogestad, S., Optimizing Control of Petyluk Distillation: Understanding the Steady-State Behavior, *Computers Chem. Eng.*, 21, Suppl., pp. S249–S254, 1997.
8. Luster, E.W., (Standard Oil Development Co.) Apparatus for Fractionating Cracked Products, U.S. Pat. 1,915,581, 1933.
9. Brugman, A.J., Process and Device for Fractional Distillation of Liquid Mixtures, More Particularly Petroleum, U.S. Pat. 2,295,256, 1942.
10. Wright, R.O., (Standard Oil Development Co.) Fractionation Apparatus, U.S. Pat. 2,471,134, 1949.
11. Knott, M., Distillation's great leap forward?, *Process Eng.*, pp. 38–34, February 1993.

EMERGENCE OF PARTITIONED COLUMNS

The advantages offered by thermally coupled columns and partitioned columns in various separation applications were already recognized in the first half of the twentieth century [8, 9, 10]. For a long time, however, no commercial columns were actually built. This reluctance was probably due to the complex calculations required for their design, as well as uncertainty about their control.

Over the past several years, however, partitioned columns have been drawing more favorable attention [11, 12, 13], with some textbooks [14] even providing elementary guidelines for their design. Research groups [5, 15] have set up laboratory-scale units to study the dynamic behavior of these systems. Meanwhile, some engineers [6, 7], have expressed grave concerns about the control of partitioned columns, while others [16] have claimed that only certain column arrangements will be "operable."

Until recently, only BASF AG accepted the partitioned-column concept wholeheartedly [17], operating several (currently 28) such columns, some for well over a decade. But increasingly, other companies have started implementing this technology [18, 19]. The successful design and operation of partitioned columns by BASF, Sasol Ltd. [20, 21] and others [22] demonstrates that stable, on-specification operation can be achieved, by using the appropriate control systems (see main text of article).

Some developmental work has been done to extend the concept in order to achieve a separation of four or more components [23], using multiple partitions within a column. To date, no serious attempts have been made to implement such a complex system.

At any rate, the advantages that partitioned columns offer will almost certainly ensure that many more processes will incorporate such units in the future. □

Acknowledgements

Linde and Sasol thank Dr. Gerd Kaibel and Dipl.-Ing. Manfred Stroezel of BASF AG for their technical advice and guidance during the implementation of the first partitioned column in the Sasol complex. Much of that know-how is reflected in this article. ■

Edited by Nicholas P. Chopey

Authors



Hans Becker is a process manager for chemical and gas plants in the process engineering contracting division of Linde AG, (Dr.-Carl-von-Linde-str. 6–14, D82049 Hellriegelskreuth, Germany; E-mail: hans.becker@linde-vn.de). Since joining Linde in 1976, he has dealt with new-process development and design of "first of its kind" plants. He has published or presented some 20 papers, and has filed numerous patents. A doctoral-degree holder, he studied technical physics at the Technical University of Munich.



Sven Godorr (E-mail: sven.godorr@linde-vn.de) is a process engineer in the same division of Linde AG, involved in process design and optimization of gas plants, including ones to manufacture synthesis gas. Previously, he was with South Africa's Sasol Technology (Pty) Ltd., responsible for conceptual development of new processes. His B.Sc., M.Sc. and PhD degrees in chemical engineering are from the University of Witwatersrand, South Africa.



Helmut Kreis (E-mail: helmut.kreis@linde-vn.de), in the same Linde division, heads the department for process design of components, for chemical and gas-processing plants. Since joining Linde in 1969, his areas of specialization have included process-industries distillation, absorption, heat transfer and fluid flow. He is a member of the VDI/GVC-Tachauschuss – Thermal Separation of Gas/Liquid Mixtures, and head of the VDI working group for chemical engineering in Munich. He holds an M.Sc. degree in mechanical engineering from the University of Munich.



James Vaughan is a technical manager in the Concept Development Dept. at Sasol Technology (P. O. Box 5486, Johannesburg 2000, South Africa; Phone: +27 11 441 2588; E-mail: james.vaughan@sasol.com). With the company since 1995, he is responsible for technical development and design of its comonomer plants. He holds a B.Sc. and a Ph.D. in chemical engineering from South Africa's University of Cape Town.

Reduce Costs with Dividing-Wall Columns

MICHAEL A. SCHULTZ,
DOUGLAS G. STEWART,
JAMES M. HARRIS,
STEVEN P. ROSENBLUM,
MOHAMMED S. SHAKUR
AND DENNIS E. O'BRIEN,
UOP

These distillation columns can significantly reduce capital and energy expenses vs. conventional multicolumn arrangements.

Distillation is the primary separation process used in the chemical processing industries (CPI). While this unit operation has many advantages, one drawback is its significant energy requirement. The dividing-wall column (DWC) offers an alternative to conventional distillation towers, with the possibility of savings in both energy and capital costs.

For example, two applications of DWC technology designed by UOP are now part of a new UOP linear alkyl benzene (LAB) complex that saved 9% of the total fractionation energy used in this complex. Based on a present world LAB production of 2.6 million m.t./yr, and a fuel value of \$10.8/million kcal, a \$12.8 million annual energy savings would be possible if these DWCS were used in place of conventional multicolumn trains in every complex.

In addition, the uninstalled equipment cost for the total complex is reduced by about 10%. The capital cost savings result from the reduction in the quantity of equipment (*i.e.*, one column, reboiler, condenser, etc., instead of two of each). There are also indirect benefits: a DWC requires less plot area and, therefore, shorter piping and electrical runs, a smaller storm runoff system and other associated benefits. The flare loads are reduced because of the smaller heat input and less fire-case surface, leading to a smaller flare system.

While a DWC is not suitable for every situation, clearly it can be an attractive alternative to conven-

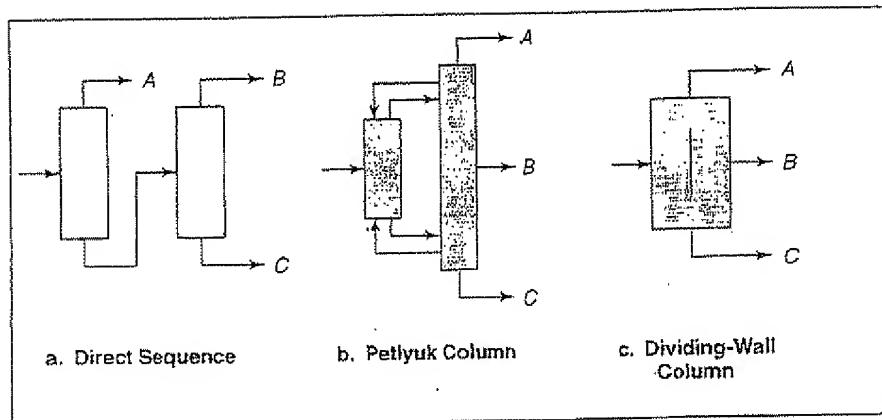
tional distillation. This article will review recent developments in DWC technology and provide guidelines for the design of these columns. Finally, two applications will be discussed to illustrate the design process.

DWC background

A DWC is not a new concept, having been introduced in 1949 (1). However, lack of reliable design methods, and concerns about the operation and control of these columns have prevented their widespread application. Work being done in both academia and industry is helping to address these concerns. Several authors provide some good background for understanding the theory behind Petlyuk columns and/or DWCS (2-11). A recap is provided here to show how a single DWC can replace an existing two-column sequence.

ABC split and Petlyuk evolution

Consider a mixture consisting of three components, *A*, *B* and *C*, where *A* is the lightest and *C* the heaviest. Figure 1a shows how this separation would be accomplished in a direct sequence of two distillation columns. For some mixtures, for instance when *B* is the major component and the split between *A* and *B* is roughly as easy as the split between *B* and *C*, this configuration has an inherent thermal inefficiency (Figure 2). In the first column, the concentration of *B* builds to a maximum at a



■ Figure 1.
Separating three components by distillation
can be done in a variety of arrangements
with one or two towers.

tray near the bottom. On trays below this point, the amount of the heaviest component *C* continues to increase, diluting *B* so that its concentration profile now decreases on each additional tray toward the bottom of the column. Energy has been used to separate *B* to a maximum purity, but because *B* has not been removed at this point, it is remixed and diluted to the concentration at which it is removed in the bottoms. This remixing effect leads to a thermal inefficiency.

Figure 1b shows a configuration that eliminates this remixing problem. This prefractionator arrangement, or Pethyuk column (11) as it is commonly known, performs a sharp split between *A* and *C* in the first column, while allowing *B* to distribute between these two streams. All of *A* and some of *B* are removed in the overhead of the smaller prefractionation column, while all of *C* and the remaining *B* are removed in the bottoms of the prefractionation column. The upper portion of the second column then performs an *A/B* separation, while the lower portion separates *B* and *C*. During the design phase, the fraction of *B* separated in the overhead of the prefractionator can be set to prevent the remixing seen in the direct sequence of Figure 1a. The thermal inefficiency has been eliminated, leading to a significant energy saving of about 30% for a typical design and can reach 50% or 60% for unconventional ones (3, 7, 12).

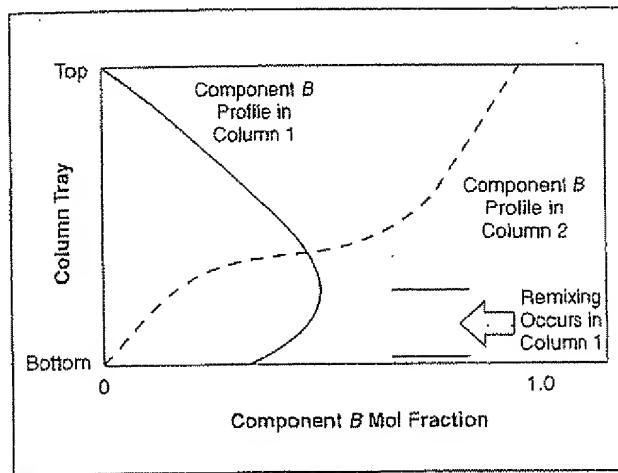
Figure 1b shows that the Pethyuk arrangement is thermally coupled. In other words, vapor and liquid streams from the second (main) column are used to provide vapor and liquid traffic in the prefractionator. This system has only one condenser and one reboiler, and both are attached to the second column. Because the Pethyuk arrangement has fewer pieces of major equipment than does the conventional two-column sequence, total capital costs may be reduced.

Integrating the prefractionation column into the same shell as the main column can further reduce the amount of equipment. This is the DWC (Figure 1c). Assuming that heat transfer across the dividing wall is

negligible, a DWC is thermodynamically equivalent to a Pethyuk column. When compared to a conventional two-column system, a capital cost savings of up to 30% is typical (3, 7, 12).

As discussed above, a DWC can be used to separate three products in a single column. In evaluating whether a DWC is a viable option, consider the thermodynamic properties, as well as the composition of the stream to be separated, in addition to the product requirements. Based on this information, some guidelines are useful to determine whether a DWC is a good candidate for accomplishing a particular separation:

- *Product purity:* the purity of the middle product is greater than can be achieved in a simple sidedraw column. Therefore, when a high-purity middle product is desired, a DWC should be considered. If strict purity specifications are not required for the middle product, a simple sidedraw column may be sufficient for the task. However, even for this case, a DWC may be advantageous as it may accom-



■ Figure 2. Remixing of Component *B* occurs in a conventional,
two-column direct-sequence arrangement.

plish the separation in a smaller column using less energy compared to the simple sidestream setup.

• *Feed composition:* Component *B* should be in excess, and components *A* and *C* should be present in fairly equal quantities. A typical rule of thumb is that a DWC is most advantageous when the feed consists of about 60–70 mol% *B*, with *A* and *C* then making up the rest of the feed in roughly equal amounts. It is important that this rule not be applied indiscriminately, though, because the relative volatility of the components is important as well. The relative volatility α_{ij} is a factor indicating the difficulty of separation of two components. The larger the value of this parameter, the easier is a separation for a given system.

• *Relative volatility:* When *B* is a significant portion of the feed, a DWC can be advantageous as long as the split between *A* and *B* is at least as difficult as that between *B* and *C* (11). When the *A/B* split is fairly easy relative to the *B/C* split, the DWC's advantages may not be great enough to justify its selection over a simple direct sequence.

• *Revamp possibilities:* To increase the throughput through an existing simple sidestream column, a dividing wall can be inserted through a portion of the column. This is an extension of the first rule concerning product purity. In this case, however, if increased throughput is desired through an existing simple-sidestream column, it may be effected by inserting a dividing wall through a portion of the column.

As with any guidelines, there are exceptions, but these can be useful during the screening process.

The case against DWCS

Although a DWC may offer the potential for a savings in both capital and energy costs, there are some situations in which two-column separations are preferable (3, 6). For instance, a DWC contains a single condenser and reboiler to provide the entire reflux liquid and boilup vapor to the column. The condenser operates at the coldest temperature required for the separation, while the reboiler operates at the hottest temperature. Compare this to the two-column sequence, in which the reboiler on the first column and the condenser on the second are at intermediate temperatures, so some of the duty can be supplied at intermediate levels. This may be advantageous for heat integration purposes, or if less-expensive intermediate duties are available.

Further, the two columns may require significantly different operating pressures for reasons that may include restrictions on overhead or bottoms temperatures, due to the available duties or restrictions on the bottoms temperature because of fear of degradation or polymerization. The flexibility of operating at distinctly different design pressures might outweigh the savings possible with a DWC. Additionally, a DWC will likely be taller and have a larger diameter than either

of the two conventional columns, and may surpass construction restrictions for a single tower. One solution to this problem is the use of high-performance trays. Such trays have a high capacity and efficiency, and can be spaced close together (as little as 300 mm), reducing the diameter and height of the DWC. Therefore, as in any design problem, it is necessary to evaluate the constraints and tradeoffs before proceeding with a more-detailed design.

Industry review

While theoretical studies have shown the economic advantages of DWCS in certain circumstances, industry has been hesitant to build these columns. One reason may be a lack of understanding of their design and control. In recent years, several academic groups have researched this area (14, 15, 16). One group set up a pilot-scale column to study controllability and operability (17, 18). This work has contributed to a better understanding of the design and control, and therefore a growing acceptance of DWCS within industry.

In 1985, BASF constructed and started up what is believed to be the first commercial DWC. BASF is also believed to be the leader in the total number of such columns in existence, with roughly 25 DWCS operating today (3).

Various consulting and engineering and construction firms offer design and construction services for DWCS. Kellogg Brown & Root has designed at least two DWCS for BP, at least one of which is used to split a light kerosene stream (13). Sumitomo Heavy Industries has been involved in designing at least six columns for undisclosed customers (the firm refers to its technology as a "Column in Column") using an onsite pilot-plant facility (19). Linde AG has recently constructed the world's largest DWC for Sasol, an estimated 107-m tall and 5-m in dia. Finally, Krupp Uhde has designed a column to remove benzene from pyrolysis gasoline for Veba Oel. A similar column by Krupp Uhde will start up for Chevron in the near future (19).

DWCS for detergent manufacture

DWC technology is suitable for use in the separation of streams in plants that produce detergents and aromatics, as well as in refining, hydroprocessing and reforming operations, among others. Two applications for detergent manufacture are discussed here to illustrate the steps involved in designing and constructing a DWC.

Once an application has been identified, the next step is to model the column. A general outline of the approach is given below. The explanation here will refer only to the simple *A, B, C* split described previously.

Steady-state modeling

The first step in studying a DWC is developing a static or steady-state simulation. This can be done using pro-

prietary software or with standard packages, such as Aspen Technology's Aspen Plus or Hyprotech's HYSYS Process. The latter do not include a basic dividing-wall column in their library of functions, although the user can build "custom" fractionation columns. This feature was used to model the DWC as a combination of individual tray sections, connected by internal vapor and liquid streams. For a conventional DWC, four column sections are needed: one for each of the sections above and below the wall, and one for each of the sections on either side of it. This DWC has five degrees of freedom (condenser, reboiler and three product streams). Typical specifications for design are three compositions plus the split-fraction of the vapor below the wall, and the split-fraction of the liquid above the wall.

Once the initial static simulation is developed and converges to the desired product specifications, the next step is to optimize the design. Column sections can be designed, similar to a conventional column, to balance the capital vs. energy cost trade-offs. This is more complicated than with a conventional column because liquid from the upper rectification section splits to either side of the wall. The reflux rate must be sufficient to satisfy the separation on either side.

As already noted, the key to the DWC advantage is that the middle component *B* is split in the prefractionation section, so that some *B* travels above the wall, while the remaining amount moves below the wall and out with the sidedraw stream. The split can be set to minimize the total reboiler-duty requirement by either adding or removing trays above or below the feed point, or changing the amount of liquid reflux that is sent to the feed-side of the column.

Consider a typical example, where *B* is roughly 60–70 mol% of the feed, *A* and *C* are present in equal proportions, and the difficulty of the split between *A* and *B* is roughly the same as that for the split between *B* and *C*. In the prefractionation section, the optimal design would result in *B* distributing so that half is recovered above the wall and the other half below the wall. If the feed deviates from any of these conditions, it may be necessary to take more than half of *B* either above or below the wall to optimize the design. Finally, both the vapor and liquid splits must be optimized, which will have a significant impact on the required reboiler duty.

Dynamic modeling

Next, a dynamic study is performed to ensure the successful commercialization of a DWC. One reason is

to develop suitable control schemes for this unconventional system. Another reason is that a dynamic study helps in understanding the column operation.

In steady-state modeling of a distillation column, thermodynamics are a key concern. The column is designed using the appropriate property package and setting the feed flowrate and composition, as well as the column operating conditions, to meet the product specifications. Different considerations are important for a dynamic model. These involve the physical processes occurring in the column and include the weir heights for the trays, devices such as trapout trays and the wall static-head considerations, plus the actual equipment required to make the column work.

The dynamic simulation should be thought of as a true operating plant. Some preliminary equipment designs are developed using the steady-state modeling. If the steady-state model has a condenser that produces product streams and reflux, the dynamic model must include the overhead exchanger, the overhead accumulator and the reflux and/or product pumps. Control instrumentation is required and must be tuned. Further, the dynamic simulation should model the hydraulic network. Control valves need to be included in the lines as appropriate, and additional valves may be modeled to account for pressure drops. The dry-tray pressure drop across the column is determined by accounting for the static head. Equivalent weir heights and tray

spacings must be included to accurately model liquid and vapor holdup when using theoretical stages.

The model must next be validated before using it to predict the column's dynamic behavior. One way of doing this is operating the dynamic model until it reproduces the results of the steady-state simulation. This "dynamic steady-state" can then serve as a baseline starting point for all subsequent testing. In reaching the dynamic steady-state, the controllers can be tuned and strip charts configured to record key process variables.

Once a dynamic model has been created, other disciplines should critique and try to improve it. Various groups such as operations, reactor design, instrumentation, etc., bring a unique perspective to the problem. After proving that the system will work, further testing should be done to determine the optimum location for the control points. Once a final design is set, the design team holds a final review meeting before anyone sizes and selects items such as rotating equipment, instrumentation and piping. The work process involves the following steps:

DWCs typically separate
a feed into three
products. The middle
one is purer than that
from a sidedraw column.

Reactions and Separations

1. The design engineer responsible for process engineering creates the static model of the process.

2. Taking the model from Step 1, the dynamic simulator is used to match the steady-state model.

3. The dynamic model includes control schemes recommended by technical services personnel and the process control specialists. Optional features are offered at this stage to determine the best approach. These options may include actions such as moving the temperature-control points to better anticipate compositional changes within the tower. By having a working model of the system, each new idea is tested and the results are saved for review.

4. The dynamic simulator is used to determine the effectiveness of the proposed control scheme. Once this model has been debugged, alternative control features and process upsets are modeled.

5. At minimum, the product quality should be disturbed to verify that the control instrumentation will adequately measure off-specification conditions. Difficulties in selecting the proper measurement/control point should be resolved.

6. The results of this exercise should be discussed with the project team during a meeting to review the process flow diagram. Prior to starting project work, the flow scheme, including all control requirements, should be fixed and explained to the team by the process control specialist. It may be necessary to modify the control scheme during this step.

7. The technology is incorporated into the commercial design as engineered specifications are developed for the plant.

Control system and instrumentation

Because a DWC is not a traditional piece of process equipment, the tools used to evaluate the control scheme should be reviewed in detail to ensure that they are appropriate for the tower. In addition, the sophistication necessary for the process model, the best simulator package, and the required amount of control scheme testing must be reviewed, as well. The dynamic simulator can be adapted to study control systems in a concise, structured and organized fashion.

Once the best control scheme is determined, decisions about the instrumentation details are important. Many questions surround the issue of liquid/vapor traffic on each side of the wall.

Direct control of the liquid to each side of the wall is preferred. All liquid from above the wall is removed from the column, and then controlled so that a known flow returns to each side of the wall. Direct control of the vapor split was not required for the applications that we modeled. If vapor-split control were needed, a special tray would have needed to be installed to handle this requirement.

Table 1. Cost savings for kerosene dividing-wall column.

	Capital Cost	Energy Cost
Two-Column Sequence	1.0	1.0
DWC	0.72	0.7

Kerosene prefractionation column

One application of a DWC within UOP is for a prefractionation of kerosene within a LAB complex. This is a full-range kerosene, containing C₇–C₁₆ hydrocarbons. The separation removes a “heart cut” of mid-range material, typically in the range of C₁₁–C₁₃. Looking at the generic layout in Figure 1, then A = C₁₀ and lighter, B = C₁₁–C₁₃ and C = C₁₄ and heavier.

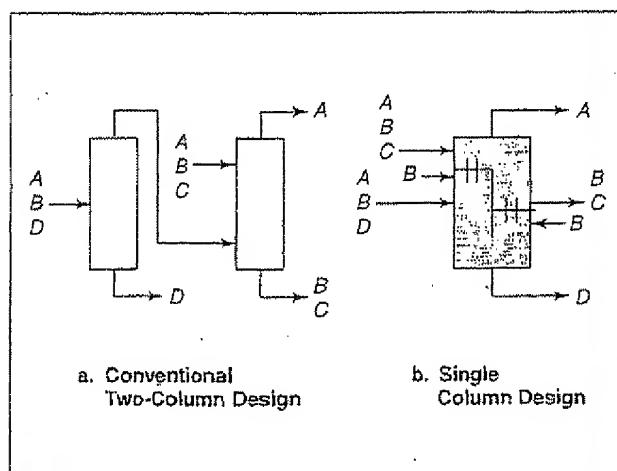
The heart-cut makes up roughly 60% of the kerosene, making this separation a good candidate for a DWC. Purity specifications are set both on the amounts of n-C₁₀ and n-C₁₄ in the middle product. A third specification is set on the recovery of n-C₁₁–n-C₁₃ in the middle product. A study found that it is not possible to successfully operate and control a DWC with purity specifications on both the light and heavy components in the sidestream product (14), which appears to preclude the use of a DWC for this application. However, this study considered distillation of pure chemical species. In each case, the light, medium and heavy key components were adjacent, meaning that there are no components that boil at temperatures in between those of the keys. Also, A, B and C represent distinct chemical species, instead of a range of species, as with kerosene prefractionation.

For the kerosene prefractionation column considered here, the feed is fractionated into three product streams. The key components are the normal paraffins in the C₁₀–C₁₄ range. Normal paraffins are desired in the range of C₁₁–C₁₃, while n-C₁₀ is the light impurity and n-C₁₄ is the heavy impurity. Hundreds of species are present that boil at temperatures between the light and middle products, and between the middle and heavy products. These distributing species are not important to the product quality, as they will be removed in a downstream separation. The column can therefore be designed so that the n-C₁₀ and n-C₁₄ will meet purity requirements.

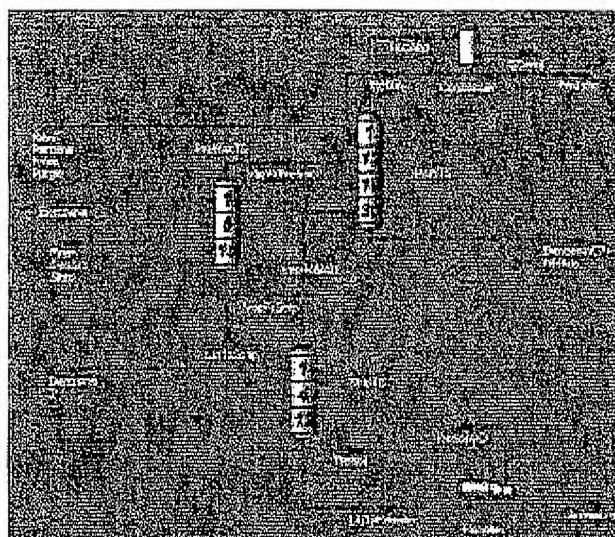
Table 1 shows the capital and energy savings that result by replacing the conventional direct sequence with a DWC in a typical system. The DWC yields an energy saving of 30% and a capital saving of 28%, both of which roughly meet the rule-of-thumb for typical DWC designs. Because of this attractive cost saving, the DWC is now a standard offering at UOP for the kerosene prefractionation column in an LAB complex.

PEP fractionation

Here, the DWC is part of the fractionation section within our Parol Enhancement Process (PEP) process



■ Figure 3. PEP fractionation via a dividing-wall column offers advantages over using two columns in series.



■ Figure 4. Screen shot of the steady-state simulation of the PEP DWC. A few changes were needed to use this model in the dynamic simulation.

KEY:

Feed Streams

Raw Pentane From Purge: Pentane (purge material) and desired C₇₊ olefin/paraffin product

Benzene, Benzene 2: Raw benzene streams used as external reflux streams

From Desorb Step: Benzene (desorbant) and heavy aromatic material

Product Streams

Pentane: Purified pentane

Benzene/C₇₊Olefins: Sent to downstream processing

Aromatics: Undesired heavy aromatic material

Equipment

PrefracTS: Feed side of the DWC

MainTS: Section above the wall, plus the section on the product side of the wall above the sidedraw stream

StripTS: Section below the wall, plus the section on the product side of the wall below the sidedraw stream

unit, a sieve separation process that removes C₇₊ aromatics from a desired C₇₊ olefin/paraffin mixture (the "C₇₊ olefins") (20). This is a batch process that uses two regenerate streams to purge and desorb the sieve beds. The regenerated effluent streams are fractionated in a DWC to purify and remove the desorbed C₇₊ aromatics and to purify and recycle pentane, one of the regenerate streams. A third product of the fractionation is a combination of the desorbant component, benzene, and "washed" C₇₊ olefins. The DWC employs a novel trap tray and external refluxing scheme to prevent the C₇₊ aromatics from mixing with the co-boiling component, the C₇₊ olefins. Figure 3 compares the conventional two-column fractionation system to the single DWC. In this figure, Component A is pentane, Component B is benzene, Component C comprises the C₇₊ olefins and Component D, the C₇₊ aromatics.

Comparing this design to a standard configuration as shown in Figure 1, a typical DWC candidate would start with a single feed stream that requires fractionation into three product streams, each with its own distinct, non-overlapping boiling range. The DWC in the PEP process has three product streams, two feeds, two external reflux streams and two co-boiling groups that must be withdrawn separately in two of the product streams. This DWC therefore did not start as a typical candidate that could follow conventional rules for such

a design. By building on previous work by others in this area, conventional design methods for DWCS were adapted to this system. Table 2 shows the economic advantage of the PEP DWC. This column uses high-performance trays, spaced at 310 mm.

Modeling the column

The steady-state model consists of prefractionation, stripping and product sections (Figure 4). When converting this model to a dynamic simulation, a few changes were necessary. First, the dividing wall actually begins just below Stage 18 of the main section and ends just above Stage 4 of the stripping section. To properly investigate the effect of the liquid split above the wall and the vapor split below the wall, it makes sense to split the main column and the bottom fractionation section each into two sections, one below the wall and one above it.

Liquid from the newly created rectification section above the wall is fed to the upper product section, simulating the total trapout tray above the wall. Vapor from the newly created stripping section splits and feeds the bottom of the prefractionation and lower-product sections. Liquid from the newly created upper product section is drawn out of the tower by a total trapout tray. This trapout tray can be modeled as a vessel with a level controller.

The generic overhead condenser is removed and replaced with an air-cooler, accumulator vessel and reflux/product pump. Other feed and product pump circuits are added. One unique aspect of the PEP separation is the cyclic nature of the feed composition. This could not be adequately modeled with a steady-state model, and, therefore, was programmed into the dynamic model. Finally, complete control instrumentation for all lines was included.

Handling upsets

Once the dynamic model was configured and validated, testing began, in which the column behavior was studied under normal and upset conditions. Offspec product must be either eliminated or minimized throughout any reasonable operation or upset. Different control configurations are tested under various upset conditions. The response to feed changes, loss of reflux, reaction to weather changes, etc., are among the upset conditions tested. As a result of this testing, a control system was designed to react quickly to upsets and be easily implemented and operated.

As already mentioned, the column design can be further optimized during the dynamic simulation study. For instance, trays can be removed from a section to study the effect on product quality. Opportunities for optimization that were not apparent from the steady-state model were identified during the dynamic study of the PEP DWC. A few items to consider beyond the product-quality goal of this column were:

- Incorporating the feed system into the model to simulate real-life upsets that are expected.
- Maintaining a reasonable heat balance.
- Evaluating the level of vaporization common during each feed upset.

The heat evaluation was also important to the success of this column, and required a study independent of all other reviews. Thermal fluctuations come from many sources. Because the feed fluctuates cyclically, a constant influence could not be anticipated. This variation causes fluctuations in the product streams, requiring a more complex model than that used in the static approach. A study evaluated the option of reboiling either side of the wall independently. This showed promise during the theoretical stage of the project, but was less attractive as the operating principles became

Literature Cited

1. Wright, R. O., U.S. Patent 2,471,134. Standard Oil Development Co., Elizabeth, NJ (May 24, 1949).
2. Ennenbach, E., et al., "Divided-Wall Columns — A Novel Distillation Concept," *Petroleum Technology Quarterly*, pp. 97–103 (Autumn 2000).
3. Becker, H., et al., "Partitioned Distillation Columns — Why, When and How," *Chem. Eng.*, 108 (1), pp. 68–74 (Jan. 2001).
4. Dünnebier, G., and C. C. Pantelides, "Optimal Design of Thermally Coupled Distillation Columns," *Ind. Eng. Chem. Res.*, 38, pp. 162–176 (1999).
5. Hernández, S., and A. Jiménez, "Design of Energy-Efficient Petlyuk Systems," *Comp. Chem. Eng.*, 23, pp. 1005–1010 (1999).
6. Lestak, F., and C. Collins, "Advanced Distillation Saves Energy and Capital," *Chem. Eng.*, 104 (7), pp. 72–76 (July 1997).
7. Triantafyllou, C., and R. Smith, "The Design and Optimisation of Fully Thermally Coupled Distillation Columns," *Trans. IChemE*, 70, Part A, pp. 118–132 (Mar. 1992).
8. Carlberg, N. A., and A. W. Westerberg, "Temperature-Heat Diagrams for Complex Columns: Part 3: Underwood's Method for the Petlyuk Configuration," *Ind. Eng. Chem. Res.*, 28, pp. 1386–1397 (1989).
9. Agrawal, R., and Z. T. Fidkowski, "Thermodynamically Efficient Systems for Ternary Distillation," *Ind. Eng. Chem. Res.*, 38, pp. 2065–2074 (1999).
10. Nikolaides, I. P., and M. M. Malone, "Approximate Design and Optimization of a Thermally Coupled Distillation Column with Prefractionation," *Ind. Eng. Chem. Res.*, 27, pp. 811–818 (1988).
11. Glins, K., and M. F. Malone, "Optimality Regions for Complex Column Alternatives in Distillation Systems," *Chem. Eng. Res. Des.*, 66, pp. 229–240 (1988).
12. Petlyuk, F. B., et al., "Thermodynamically Optimal Method for Separating Multicomponent Mixtures," *Int. Chem. Eng.*, 5, pp. 555–561 (1965).
13. Parkinson, G., et al., "The Divide in Distillation," *Chem. Eng.*, 106 (4), pp. 32–35 (Apr. 1999).
14. Halvorsen, I. J., and S. Skogestad, "Optimal Operation of Petlyuk Distillation: Steady-State Behavior," *J. Process Control*, 9, pp. 407–424 (1999).
15. Wolff, E. A., and S. Skogestad, "Operation of Integrated Three-Product (Petlyuk) Columns," *Ind. Eng. Chem. Res.*, 34, pp. 2094–2103 (1995).
16. Serra, M., et al., "Control and Optimization of the Divided Wall Column," *Chem. Eng. and Proc.*, 38, pp. 549–562 (1999).
17. Abdul Mutalib, M. I., and R. Smith, "Operation and Control of Dividing Wall Distillation Columns: Part 1: Degrees of Freedom and Dynamic Simulation," *Trans. IChemE*, 76, Part A, pp. 308–318 (1998).
18. Abdul Mutalib, M. I., et al., "Operation and Control of Dividing Wall Distillation Columns: Part 2: Simulation and Pilot Plant Studies Using Temperature Control," *Trans. IChemE*, 76, Part A, pp. 319–334 (1998).
19. Parkinson, G., "Drip and Drop in Column Internals," *Chem. Eng.*, 107 (7), pp. 27–31 (July 2000).
20. Schultz, M. A., et al., "Design and Control of a Dividing Wall Distillation Column for the Fractionation Section in the Parcol Enhancement Process (PEP)," paper presented at 2001 Spring AIChE National Meeting, Houston, TX (Apr. 22–26, 2001).

more apparent. A single reboiler with a stable heat source was selected as the best way to meet the requirements of this fractionation. As proposed, the design adjusts the vapor to the wall indirectly by manipulating the liquid traffic on each side of it. A small upset in the differential pressure across the trays will make the necessary vapor flowrate adjustment. The major benefit of this is the column's ability to meet the challenges imposed by variable feeds.

Shell fabrication and tray design

Adding a vertical partition to a conventional distillation column presents some challenges to fabricating the shell and trays. If the temperature gradient across the wall is too great, it may be necessary to install an insulated wall to prevent heat transfer that could affect the separation. Additional manways may be needed in a DWC, so that the column is accessible on either side of the wall.

The wall within the DWC effectively changes the tray design, creating two distinct noncircular sections. Also, if the wall is not exactly in the center of the column, the tray design becomes nonsymmetrical. A high-performance tray is used and is suited for this application because it has a 90-deg. tray-to-tray orientation that makes designing easy within the noncircular section and asymmetrical sections. The tray is designed for uniform flow distribution across the tray deck, which is important when the section is noncircular.

Future thoughts and conclusions

Advances in the theory of design, control and operation of a DWC have contributed to a better understanding of these columns and have led to commercial developments. As the base of commercial experience continues to grow, the number of applications should increase for both conventional and unconventional cases. Computer models will be an important part of this process, as advanced tools allow a more-complete analysis of these columns.

Chemical engineers should look for unconventional applications of DWC technology. Simply because a fractionation system does not meet the single-feed, three-key-component, three-product criteria, does not mean that DWC technology cannot be adapted to the separation. The UOP application for the PEP DWC is an example of this.

As academic research improves the understanding of dividing-wall columns, and industry finds new applications, these columns should become more common in the plant. Although it took roughly 50 years for the DWC to gain limited acceptance by some companies, perhaps the next 50 years will be a time when the DWC becomes a standard piece of equipment in the CPI.



MICHAEL A. SCHULTZ is a process research specialist in the UOP Engineering Science Skill Center (25 East Algonquin Road, Des Plaines, IL 60017-5017; Phone: (847) 375-7893; Fax: (847) 375-7904; E-mail: MASCHULT@UOP.com). He has four years of experience with UOP in a variety of process research projects. His work focuses on technical and economic analysis, process development, and process optimization of both developing and existing process technologies. He also serves as a resource for the assessment and application of UOP's dividing-wall column technology, as well as for other fractionation issues. Schultz holds a BS in chemical engineering from the Univ. of Michigan-Ann Arbor, and a PhD in chemical engineering from the Univ. of Massachusetts-Amherst. He is a member of AIChE.

DOUGLAS G. STEWART is a senior design engineer in the UOP Engineering Dept. (Phone: (847) 391-2904; E-mail: DGSTEWAR@UOP.com). He has 24 years of experience with UOP, including field service and engineering dept. assignments in a variety of UOP process technologies. He currently is involved in UOP's detergent process technologies. Stewart holds a BS in chemical engineering from the Univ. of Minnesota.

JAMES W. HARRIS is a process control coordinator in the UOP Technical Services Dept. Skill Center (Phone: (847) 391-1366; E-mail: JW.HARRIS@UOP.com). He has 12 years of experience with UOP, including field service and engineering dept. assignments in a variety of UOP process technologies. He is a technical leader for safety-instrumented-system applications; additional activities include supporting training for new-hire instrument engineers, interfacing with field-service personnel on technical issues, maintaining company procedures for specifying instrumentation, and developing new procedures to sustain technical leadership of the company. Harris holds a BS in chemical engineering from SUNY-Buffalo. He is a member of AIChE, as well as ISA, and is an active member of the ISA SP84 committee and API's subcommittee on instrumentation and control systems.

STEVEN P. ROSENBLUM is a senior process modeling specialist in the UOP Engineering Dept. (Phone: (847) 375-7182; E-mail: SPROSENB@UOP.com). He has 25 years of experience in the CPI with the last five at UOP. During his tenure at UOP, he has undertaken advanced process control and optimization, and engineering dept. assignments in a variety of UOP process technologies. He serves as an applications-support group leader for all outside vendor simulation software. Rosenblum holds a BS in chemical engineering from Cornell Univ. and an MS in chemical engineering from Northeastern Univ.

MOHAMED S. M. SHAKUR is a product manager in the process equipment group of UOP (Tonawanda, NY; Phone: (716) 879-7601; E-mail: MSSHAKUR@UOP.com). He has 23 years of experience with UOP, mainly in the distillation design group, focusing on the design and commercialization of column internals, column startup, troubleshooting and optimization. He markets UOP column internals worldwide and develops new products to complete the UOP product portfolio of high-performance column internals. Shakur holds a BEng in chemical engineering from the City College of New York.

DENNIS O'BRIEN is a detergents technology specialist in the UOP Engineering Dept. (Phone: (847) 391-2802; E-mail: DEOBRIEN@UOP.com). He has 31 years of experience with UOP, in the field service, engineering and computer departments, with assignments in a variety of UOP process technologies. He currently is a technical specialist for detergents and provides engineering support for adsorbents. O'Brien holds a BS in chemical engineering from the Univ. of Tulsa, and a MBA from Roosevelt Univ. He is a member of AIChE.